# Synthesis of 4-iodonitrobenzene

# Aims

The experiment aims to synthesis 4-iodonitrobenzene from 4-nitroaniline.

## Introduction

The synthesis began with the diazotisation of 4-nitroaniline by NaNO2 in a sulphuric acid solution. The diazonium ion is then replaced by aromatic substitution with an iodide ion from sodium iodide. This also results in the evolution on N2 gas.

## Experimental Procedure

A solution of 4-nitroaniline(1.25g, 0.0091 moles) in 1ml 90% concentration H2SO4 and 10ml deionised water was prepared. The mixture was warmed on a hot plate until the solid was fully dissolved. The mixture was cooled in an ice bath while being continuously stirred to a temperature of <5oC. A solution of 0.7g(0.0101 moles) sodium nitrite in 2ml deionised water was added to the 4-nitroaniline solution in small portions with continuous swirling. Some heat evolved during the gradual mixing process. The contents of the reaction vessel was not allowed to rise above 10oC by placing the vessel in an ice bath for the duration of the heat evolution.

 A solution of NaI(2.25g, 0.01773 moles) in 10ml deionised water was prepared separately.

This solution was added to the previously diazotised solution in a large reaction vessel. The reaction produced a brown solid product. The evolution of Nitrogen gas was observed to create foam within the reaction vessel. The product was collected via suction filtration to yield 3.51g(0.0141 moles) 4-iodonitrobenzene. The percentage yield of product collected was 155%. A 1.12g sample of the product was recrystallised from 30ml ethanol to yield 0.48g recrystallised product.

# Results

The synthesis of 4-iodonitrobenzene was successfully carried out, albeit with an inaccuracy in the apparent percentage yield.

# Discussion and Conclusions

The product acquired by suction filtration had a percentage yield of 155%. This inaccuracy could be at least partially contributed to the product being damp when wighed.

However, the recrystallisation of a sample of the final product yielded mass of less than half the sample used.

 Had recrystallisation been carried out for the entirety of the yielded product, it could be assumed(using the same ratio of masses in the recrystallisation of a sample) that the yield would have been 66.4%. Considering impurities and moisture present in the unrefined product, this projected percentage yield is more representative of the amount of 4-iodonitrobenzene present.

# Post Practical Questions

1. The percentage yield of product collected was 155%. However the projected yield after recrystallisation based on a sample was 66.4%, which would have been more accurate.
2. Heating a diaxonium salt in its aquoes acidic solution converts the salt to a phenol. Heating the diazonium salt in the experiment before the addition of the sodium iodide would have yielded p-Nitrophenol.

(i) $-bromonitrobenzene could have been synthesised from 4-nitroaniline by putting the nitroaniline in a solution of NaNO2­ and HBr, again keeping the solution below 10oC during the diazotisation process.

 (ii)4-nitrophenol could have been synthesised from the diazonium salt used in this experiment by boiling the Diazonium salt in this experiment and steam distilling out the product.

 (iii)4-nitrophenylhydrazine could be synthesised by the reduction of the diazonium salt with NaHSO3.

1. Nitration, followed by reduction of benzene yields aniline:



Bromination of the aniline yields the tri-bromo derivative:



The NH2­ is then removed as follows:

The NH2 is diazotised with of NaNO2­ in HCl. The Diazonium ion is replaced with H using H3PO2­.

